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(54) 【発明の名称】 電界発光素子用蛍光体の製造方法

(57) 【要約】

【目的】 2段焼成して電界発光素子用蛍光体を形成するにあたり、蛍光母体粒子の粒径及び形状を均一に成長させ、蛍光体の輝度向上及び長寿命化を実現する電界発光素子用蛍光体の製造方法を提供する。

【構成】 蛍光母体に付活剤と共付活剤兼用粒成長促進剤とを添加した混合物を高温で長時間一次焼成し、蛍光母体粒子を所定径に成長させて中間蛍光体を得た後、それを二次焼成して蛍光体を形成するにあたり、上記一次焼成時及び／又は二次焼成時にハロゲン化合物以外のマグネシウム化合物を蛍光母体1molに対し0.1~10mol%添加する。

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## 【特許請求の範囲】

【請求項1】 蛍光母体に付活剤と共付活剤兼用粒成長促進剤とを添加した混合物を高温で一次焼成し、蛍光母体粒子を所定径に成長させて中間蛍光体を得た後、それを二次焼成して蛍光体を形成するにあたり、上記一次焼成時及び／又は二次焼成時にハロゲン化物以外のマグネシウム化合物を蛍光母体1molに対し0.1～10mol%添加することを特徴とする電界発光素子用蛍光体の製造方法。

【請求項2】 一次焼成後の中間蛍光体に静的又は動的に外力を加えた後、二次焼成することを特徴とする請求項1記載の電界発光素子用蛍光体の製造方法。

【請求項3】 蛍光母体は硫化亜鉛、付活剤は銅化合物、そして粒成長促進剤は塩化マグネシウムを含むハロゲン化物であることを特徴とする請求項1及び2記載の電界発光素子用蛍光体の製造方法。

## 【発明の詳細な説明】

【0001】

【産業上の利用分野】 本発明は、特に分散型電界発光素子の発光層等に使用される蛍光体の製造方法に関するものである。

【0002】

【従来の技術】 液晶ディスプレイパネルのバックライト等に使用される有機分散型電界発光灯（以下、ELパネルと称す）を図2（a）（b）を参照して以下に示す。上記ELパネル（1）は、背面電極（2）と反射絶縁層（3）と発光層（4）と透明電極（5）とを順次、積層して形成された電界発光素子（6）の上下にポリアミド樹脂等の吸湿フィルム（7）を配置し、吸湿フィルム（7）を含む電界発光素子（6）の全体をフッ素系樹脂等からなる外皮フィルム（8）で気密に封止すると共に、背面電極（2）及び透明電極（5）から外皮フィルム（8）の封止部位を通してリード（9）（10）を導出したものである。

【0003】 上記発光層（4）は、図2（c）に示すように、有機バインダ（11）に、銅で活性化した硫化亜鉛（ZnS）等の蛍光体（12）を分散させることによって形成され、有機バインダ（11）により反射絶縁層（3）に接着されている。上記ELパネル（1）では、リード（9）（10）から背面電極（2）と透明電極（5）間に高電圧を印加することによって、両電極（2）（5）間に挟まれた発光層（4）の蛍光体（12）を発光させ、所望の発光輝度で駆動させている。

【0004】 上記蛍光体（12）は、一般的に粒状硫化亜鉛（ZnS）を蛍光母体とし、それに銅化合物（CuSO<sub>4</sub>）からなる付活剤、及び塩化物（MgCl<sub>2</sub>、NaCl、SrCl<sub>2</sub>）からなる共付活剤兼用粒成長促進剤（フラックス）を添加した混合粉末を焼成して得られる。そして、1～3μm径の蛍光母体原料をフラックスにより20～30μm径まで粒成長させて長寿命化を図

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ると共に、銅と塩素を発光中心としてドーピングし、高輝度化を図る。

【0005】 そこで、上記蛍光体（1）を形成する際、まずZnS（蛍光母体）1molに対してCuSO<sub>4</sub>（付活剤）を0.1～0.2mol%添加して乾燥処理する。次に、図2（d）に示すように、MgCl<sub>2</sub>の3mol%と、NaClの3mol%と、SrCl<sub>2</sub>の3mol%とを混合してなるフラックス（12a）を蛍光母体（12b）の微粉末と共にルツボ（13）内に供給する。そして、ルツボ（13）内で上記混合粉末を1150℃で6時間、加熱して一次焼成すると、まずフラックス（12a）（融点700℃）が溶融して液状となり、ルツボ内の底部付近に溜まって逐次、蒸気となる。そこで、ルツボ内を攪拌すると、フラックス（12a）が分散すると共に、その融液又は蒸気を介して蛍光母体粒子が互いに溶融・結合し合って次第に粒径が大きく成長する。上記一次焼成後、水洗してフラックス分を除去し乾燥させて中間蛍光体を形成すると、それをラバープレス等にて静的に加圧する。次に、700℃で1時間、二次焼成（アニール）し、更に、酸（HCl）及びシアン（KCN）洗浄して蛍光体表面の余剰の銅化合物を除去した後、水洗してシアン成分を除去し、乾燥させて分級（ふるい分け）すると、所望の電界発光素子用蛍光体を得る。

【0006】

【発明が解決しようとする課題】 解決しようとする課題は、一次焼成時のフラックス量が蛍光母体1molに対して10mol%以下であるため、フラックス（12a）が溶融すると、ルツボ内で蒸気になり、蛍光母体（12b）の粒子を覆いつつ結合し合って粒成長し、フラックス蒸気による固相反応が主体となる。フラックス蒸気が存在している領域では、粒子が十分に成長する一方、フラックス蒸気が不十分であると、粒子の未成長や不十分な成長粒子も生じ、成長した粒子が不揃いになって粒度分布が広くなり、輝度や寿命が低下したり、或いは収率が低下し、又、粒形状が不安定になって電界が加わり難くなるという不具合が生じる。又、固相反応によれば、異なる結晶軸を持つ粒子が結合してそのまま粒成長し易いため、それにより粒子の各結晶軸方向によって電界の加わり方が異なり、蛍光母体（12b）として輝度が低下するという不具合もある。この場合、高温で長時間、焼成すれば、粒子の各結晶軸方向が揃い易くなるが、焼成条件を無闇に変えることは出来ず、實際上、実施不能である。

【0007】

【課題を解決するための手段】 本発明は、蛍光母体に付活剤と共付活剤兼用粒成長促進剤とを添加した混合物を高温で一次焼成し、蛍光母体粒子を所定径に成長させて中間蛍光体を得た後、それを二次焼成して蛍光体を形成するにあたり、上記一次焼成時及び／又は二次焼成時に

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ハロゲン化物以外のマグネシウム化合物を蛍光母体1molに対し0.1~10mol%添加することを特徴とする。

【0008】

【作用】上記技術的手段によれば、蛍光母体の粒成長反応の際、マグネシウム化合物の添加により未成長粒子の発生が消滅し、粒径及び形状が揃って安定化する。

【0009】

【実施例】本発明に係る電界発光素子用蛍光体の製造方法の実施例を図1(a)(b)を参照して以下に説明する。本発明の特徴は、2段焼成により電界発光素子用蛍光体を形成するにあたり、一次焼成時及び/又は二次焼成時にフラックス用ハロゲン化物以外のマグネシウム化合物、例えば $MgSO_4$ （硫酸マグネシウム）を蛍光母体1molに対し0.1~10mol%添加することである。上記フラックスとしては、アルカリ金属やアルカリ土類金属のハロゲン化物を複数種類、選択的に使用すれば良い。

【0010】上記手段に基づき本発明を説明する。まず $ZnS$ （蛍光母体）の1molに対して $CuSO_4$ （付活剤）を0.1~0.2mol%添加して乾燥処理し、且つ、 $MgSO_4$ を0.1~10mol%添加して湿式又は乾式により混合する。次に、図2(d)と同様に、 $MgCl_2$ 10mol%及び $NaCl$ 1mol%を混合してなるフラックス（付活剤兼用粒成長促進剤）（12a）を硫酸マグネシウム添加の蛍光母体（12b）の微粉末と共にルツボ（13）内に供給する。そして、ルツボ（13）内で上記混合粉末を1100°Cで3時間、大気雰囲気中で加熱及び攪拌して一次焼成する。上記一次焼成後、水洗して残留フラックス分を除去し、120°Cで12時間、加熱して乾燥させ、中間蛍光体を形成すると、それをボールミル等により動的に加圧して外力を加える。次に、750°Cで3時間、アニール（二次焼成）した後、酸（ $HCl$ ）及びシアン（ $KCN$ ）洗浄する。そして、120°Cで12時間、加熱して乾燥させ、分級して所望の電界発光素子用蛍光体を得る。

【0011】上記実施例によれば、一次焼成時の蛍光母体（12b）の粒成長反応の際、硫酸マグネシウムを添加することにより未成長や不足成長粒子の発生が消滅し、粒径及び形状が揃って安定化する。又、一次焼成後に外力を加えることにより中間蛍光体粒子の内部に歪みが生じ、更に、二次焼成によって粒子内で均一に分散してい

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た銅が移動して歪みの部分に集まり、粒子内に導電層が生じ（偏析）、発光中心（銅、塩素）が効率良く電界発光する。この時、粒径及び形状が均一に揃っているため、外力を加える際、粒子に一個ずつ均等に応力が加わって上記歪みが均等に形成され、輝度向上に寄与する。

【0012】ここで、図1(a)に示す粒度分布測定グラフによれば、本発明の硫酸マグネシウム添加時における粒度分布図（A）（破線）は、従来の硫酸マグネシウム未添加時における粒度分布図（B）（実線）に比べて先鋭になり、分布が狭くなることが知られる。又、図1(b)に示す輝度測定グラフによれば、本発明実施例において輝度は、従来よりも10~25%向上することが知られる。

【0013】尚、上記マグネシウム化合物としては、硫酸マグネシウムの他、酸化マグネシウムや硝酸マグネシウム等も好適である。又、一次焼成時に代えて二次焼成時にマグネシウム化合物を所定量添加しても同様の効果を得ることが出来、又、一次及び二次焼成時の両方に添加しても良い。この場合、各焼成時の添加量をやや減らし、その総量を単独の添加量に適合させれば良い。

【0014】

【発明の効果】本発明によれば、2段焼成して電界発光素子用蛍光体を形成する際、一次焼成時及び/又は二次焼成時にハロゲン化物以外のマグネシウム化合物を蛍光母体1molに対し0.1~10mol%添加したから、蛍光母体の粒成長反応において粒径及び形状が均一に揃い、輝度向上及び長寿命化を図ることが出来、又、粒度分布が狭くなって収率が向上する。

【図面の簡単な説明】

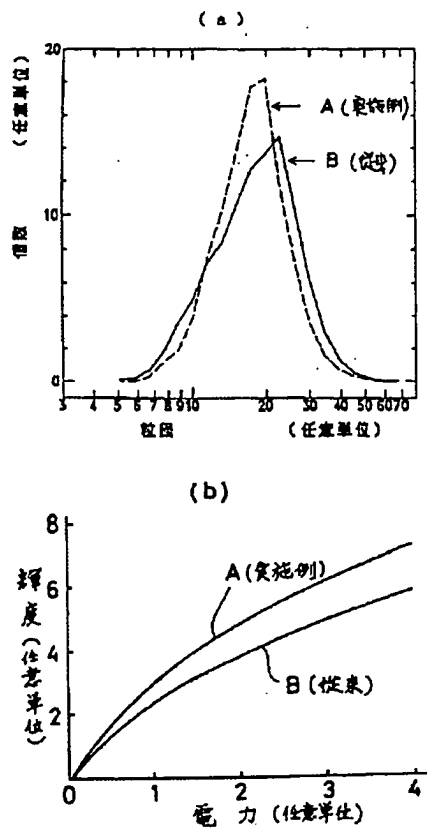
【図1】(a)は本発明に係る電界発光素子用蛍光体の製造方法による粒度分布グラフである。(b)は本発明に係る電界発光素子用蛍光体の製造方法による電力に対する輝度分布グラフである。

【図2】(a)は有機分散型電界発光灯の一例を示す側断面図である。(b)は有機分散型電界発光灯の一例を示す平面図である。(c)は発光層の部分側断面図である。(d)は従来の電界発光素子用蛍光体の製造方法の実施例の一工程を示すルツボの側断面図である。

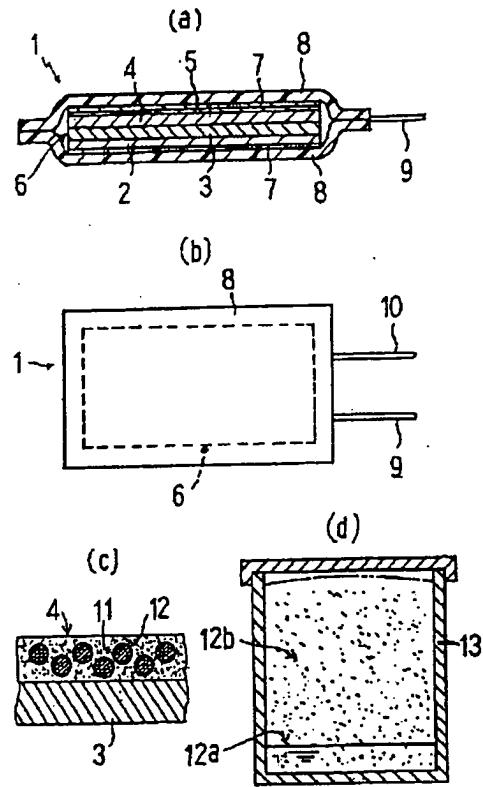
【符号の説明】

12a 粒成長促進剤  
12b 蛍光母体

【図1】



【図2】



(19) The Patent Office in Japan (JP)

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(54) [Name of invention] Manufacturing method of phosphors for the use of the electro luminescent element

(57) [Summary]

[Purpose] It is to provide the manufacturing method of the phosphors for the use of the electro luminescent element, which shall actualize the improvement of the luminance and longer life of the phosphors by evenly making the particle size and the shape of the

phosphor particles grow when forming the phosphor for the filed emitting element by the 2-step firing process.

[Structure] In order to create the phosphor by the second firing after obtaining the intermediate phosphor by growing the phosphor host material particle to the designated size from the initial firing, which is made to the mixture of the activator and the activator with the particle growth accelerating agent added phosphor, at the high temperature for long hours, approximately 0.1 to 10 mol % of the magnesium compound excluding the halide per 1 mol of the phosphor host material shall be added during the abovementioned initial firing and/or the second firing.

[Range of the patent claim]

[Claim 1] It is a manufacturing method of the phosphor for the use of the electro luminescent element, which shall be characterized by creating the phosphor by the second firing after obtaining the intermediate phosphor by growing the phosphor host material particle to the designated size from the initial firing, which is made to the mixture of the activator and the activator with the particle growth accelerating agent added phosphor, at the high temperature for long hours, approximately 0.1 to 10 mol % of the magnesium compound, excluding the halide per 1 mol of the phosphor host material, shall be added during the abovementioned initial firing and/or the second firing.

[Claim 2] It is a manufacturing method of the phosphor for the use of the electro luminescent element which is mentioned in Claim 1, which shall be characterized by performing the second firing after the outer power shall be added statically or dynamically to the intermediate phosphor which the initial firing was performed to.

[Claim 3] It is a manufacturing method of the phosphor for the use of the electro luminescent element, which is mentioned in Claim 1 and 2, which shall be characterized by the phosphor host material being the zinc sulfide, the activator being the copper compound and the particle growth accelerating agent being halide, which shall contain magnesium chloride.

[Detailed explanation of the invention]

[0001]

[Utility field of the invention]

This invention is concerning the manufacturing method of the phosphor, which shall be used especially for the emission layer, etc. of the scattered style filed emitting element.

[0002]

[Existing technique]

The organic scattered style electro luminescent light, which shall be used for the back light, etc. of the display panel of the liquid screen (hereinafter referred to as EL panel) shall be indicated below referring to (a) and (b) of Figure 2. The abovementioned EL panel (1) is made by arranging the absorbing films (7) of polyamide resin, etc. on top and the bottom of the electro luminescent element (6), which is composed of the laminated layers of the rear electrode (2), the reflecting insulation layer (3), the emission layer (4)

and the transparent electrode (5) in sequence, and tightly sealing the whole part of the electro luminescent element (6), which shall include the absorbing film (7) using the skin film (8), which is made of the fluoride resin, etc., and deriving the leads (9) and (10) through the sealing part of the skin film (8) from the rear electrode (2) and the transparent electrode (5).

[0003] The abovementioned emission layer (4) shall be created by scattering the phosphor (12) of zinc sulfide (ZnS), etc., which is activated by copper, to the organic binder (11), which is shown in (c) of Figure 2, and it is bonded to the reflecting insulation layer (3) by the organic binder (11). The abovementioned EL panel (1) shall drive at the desired emitting luminance by making emission from the phosphor (12) of the emission layer (4), which is sandwiched between the two electrodes of (2) and (5), by applying the high voltage between the rear electrode (2) and the transparent electrode (5) from the leads (9) and (10).

[0004] The abovementioned phosphor (12) generally shall use the particle style zinc sulfide (ZnS) as the phosphor host material and shall be obtained by firing, which shall be performed to the mixture powder, which the activator, which is made of the copper compound ( $\text{CuSO}_4$ ), and the activator with the particle growth accelerating agent (flux), which is made of chloride ( $\text{MgCl}_2$ ,  $\text{NaCl}$ ,  $\text{SrCl}_2$ ), are added to. And by making the particle growing to be the particle size of 20 to 30  $\mu\text{m}$  by flux using the phosphor host raw material of the particle size of 1 to 3  $\mu\text{m}$ , the longer life can be obtained, and by doping copper and chloride as the main emitting material, higher luminance can be obtained.

[0005] When creating the abovementioned phosphor material (1), 0.1 to 0.2 mol % of  $\text{CuSO}_4$  (activator) shall be added per 1 mol of ZnS (phosphor host material), and then the dry treatment shall be performed. Next, as shown in (d) of Figure 2, flux (12a), which is composed of the mixture of 3 mol% of  $\text{MgCl}_2$ , 3 mol% of  $\text{NaCl}$  and 3 mol% of  $\text{SrCl}_2$ , and the fine powder of the phosphor host material (12b) shall be supplied to the inside of the crucible (13). And when the initial firing is applied to the abovementioned powder mixture by heating for 6 hours at 1150  $^{\circ}\text{C}$  in the crucible (13), first the flux (12a) (melting point 700  $^{\circ}\text{C}$ ) shall become liquid by melting, and it shall gather around the bottom area of the crucible, then it shall become vapor accordingly. At this time, if the inside of the crucible is stirred, then the flux (12a) shall be scattered and the particle size shall grow larger gradually by melting and then combining of phosphor host material particles through the melted liquid or vapor. After the abovementioned initial firing, the intermediate phosphor shall be created by drying after washing off by water to remove the flux, then the pressure shall be applied to this by rubber press, etc. statically. Next, the second firing (anneal) shall be performed for one hour at 700  $^{\circ}\text{C}$ , then acid ( $\text{HCl}$ ) and cyan ( $\text{KCN}$ ) rinsing shall be performed to remove the excess copper compound of the surface of the phosphor, and then the cyan component shall be removed by washing using water, and after that, the desired phosphor for the electro luminescent element can be obtained by classifying (screening) after the drying procedure has completed.

[0006]

[Problem to be solved by the invention] The issue which needs to be solved is that because the flux amount when the initial firing is performed is less than 10 mol% per 1 mol% of the phosphor host material, when the flux (12a) melts, it shall become the vapor in the crucible, and the particle size shall grow by the melted flux covering the particle of the phosphor host material (12b) in order to combine, and the solidus reaction shall become the main part by the flux vapor. On one hand, the particles shall grow sufficiently within the area where the flux vapor shall exist, however, on the other hand, if the flux vapor is not enough, then the particles might not grow or might not grow sufficiently, which shall create the uneven particles, which shall expand the particle size distribution, which might bring the result of decreased luminance and life, or unstable particle shapes, which makes it difficult for the electric field to apply to. Also, according to the solidus reaction, it is easy to make the particles grow just by combining the particles of the different crystal shaft, which makes the applying of the electric field different depending on each direction of the crystal shaft of the particles, which shall reduce the luminance of the phosphor host material (12b). In this case, if the firing is performed at the high temperature for long hours, each direction of the crystal shaft of the particles can be the same, however, it is not possible to change the firing condition thoughtlessly, and therefore, in an actual base, it is impossible to perform this.

[0007]

[Method of how to solve the problem] This invention shall be characterized by creating the phosphor by the second firing after obtaining the intermediate phosphor by growing the phosphor host material particles to the designated size from the initial firing, which is made to the mixture of the activator and the activator with the particle growth accelerating agent added phosphor, at the high temperature for long hours, approximately 0.1 to 10 mol % of the magnesium compound excluding the halide per 1 mol of the phosphor host material shall be added during the abovementioned initial firing and/or the second firing.

[0008]

[Function] Concerning the abovementioned technical method, non-growing particle shall not occur by adding the magnesium compound when making particle growth reaction of the phosphor host material, and the particle size and the shape shall become even and stable.

[0009]

[Example of implementation] The implementation example of the manufacturing method of the phosphor for the electro luminescent element, which is related to this invention, shall be explained below referring to (a) and (b) of Figure 1. The characteristics of this invention is that in order to create the phosphor for the filed emitting element by the 2-step firing, 0.1 to 10 mol% of the magnesium compound for flux except halide, for example  $\text{MgSO}_4$  (magnesium sulfide), shall be added per 1 mol% of the phosphor host material. As for the abovementioned flux, several kinds of alkaline metals or halide of the alkaline earth metals can be used selectively.

[0010] This invention shall be explained in accordance with the abovementioned method. First, 0.1 to 0.2 mol % of  $\text{CuSO}_4$  (activator) shall be added per 1 mol of  $\text{ZnS}$  (phosphor host material), and then the dry treatment shall be performed, and further, 0.1 to 10 mol% of  $\text{MgSO}_4$  shall be added and then mixed either by wet style or dry style. Next, as shown in (d) of Figure 2, flux (activator with the particle growth accelerating agent) (12a), which is composed of the mixture of 10 mol% of  $\text{MgCl}_2$  and 11 mol% of  $\text{NaCl}$ , and the fine powder of the phosphor host material, which magnesium sulfide is added to, (12b) shall be supplied to the inside of the crucible (13). And the initial firing is applied to the abovementioned powder mixture, which shall be stirred and heated within the ordinary atmosphere for 3 hours at  $1100^\circ\text{C}$  in the crucible (13). After the abovementioned initial firing, the intermediate phosphor shall be created by heating to make it dry for 12 hours at  $120^\circ\text{C}$  after washing off by water to remove the remaining flux, then the pressure shall be applied to this by the ball mill method, etc. dynamically in order to add the outer power. Next, the second firing (anneal) shall be performed for 3 hours at  $750^\circ\text{C}$ , then acid ( $\text{HCl}$ ) and cyan ( $\text{KCN}$ ) rinsing shall be performed. And then heating shall be performed to make it dry in order to obtain the desired phosphor for the electro luminescent element by classifying (screening).

[0011] Concerning the abovementioned technical method, non-growing particles shall not occur by adding the magnesium sulfide when making the particle growth reaction of the phosphor host material (12b) during the initial firing process, and the particle size and the shape shall become even and stable. Also, by adding outer power after the initial firing has completed, it generates the warp inside of the intermediate phosphors, and further, by performing the second firing, the copper, which was scattered evenly within the particles, shall be moving to the warp area, which shall generate the conductive layer (segregation), and the main emitting material (copper and chlorine) shall emit efficiently. At this point, due to the reason that the particle sizes and the shapes are even, when the outer power is added, even stress shall be added to each particle and the abovementioned warp shall be evenly formed, which shall contribute for the improvement of the luminance.

[0012] According to the particle size distribution graph, which is shown in (a) of Figure 1, the particle size distribution figure of when magnesium sulfide of this invention is added to, which is indicated in (A) (dashed line), shall become radical compared to the particle size distribution figure of the existing case of when magnesium sulfide is not added to, which is indicated in (B) (solid line), and it is known to have narrower distribution. Also, according to the luminance measurement graph, which is shown in (b) of Figure 1, the luminance of the implementation example of this invention shall be improved by 10 to 25% compared to the existing one.

[0013] Further, as to the abovementioned magnesium compound, magnesium oxide and magnesium nitrate shall also be suitable besides magnesium sulfide. Also, even if the designated amount of the magnesium compound is added during the second firing instead of the initial firing, the same effect can be obtained, and also it can be added to during both the initial and the second firing processes. In this case, adding amount of each firing

process shall be reduced by little, and the total amount can be equal to the designated adding amount to one firing process.

[0014]

[Effectiveness of the invention] Concerning this invention, when creating phosphor for the electro luminescent element by 2-step firing processes, because approximately 0.1 to 10 mol % of the magnesium compound excluding the halide per 1 mol of the phosphor host material shall be added during the abovementioned initial firing and/or the second firing, the particle size and the shape shall become even, which improved luminance and long life, and narrowed the particle size distribution.

[Simple explanation of the figures]

[Figure 1] (a) is a particle size distribution graph using the manufacturing method of the phosphor for the electro luminescent element, which is related to this invention. (b) is a luminance distribution graph against the electric power concerning the manufacturing method of the phosphor for the electro luminescent element, which is related to this invention.

[Figure 2] (a) is a side cross section figure to indicate one example of the organic scattered style electro luminescent light. (b) is a ground plan to indicate one example of the organic scattered style electro luminescent light. (c) is a partial side cross section figure of the emission layer. (d) is a side cross section figure of the crucible, which shall indicate one process of the implementation example of the manufacturing method of the phosphor for the electro luminescent element.

[Explanation of symbols]

12a: Particle growth accelerating agent

12b: Phosphor host material

[Figure 1]

(a)

Vertical: Number of pieces (voluntary unit)

Horizontal: Particle size (voluntary unit)

A: Implementation example

B: Existing model

(b)

Vertical: Luminance (voluntary unit)

Horizontal: Electric power (voluntary unit)

A: Implementation example

B: Existing model

[Figure 2]